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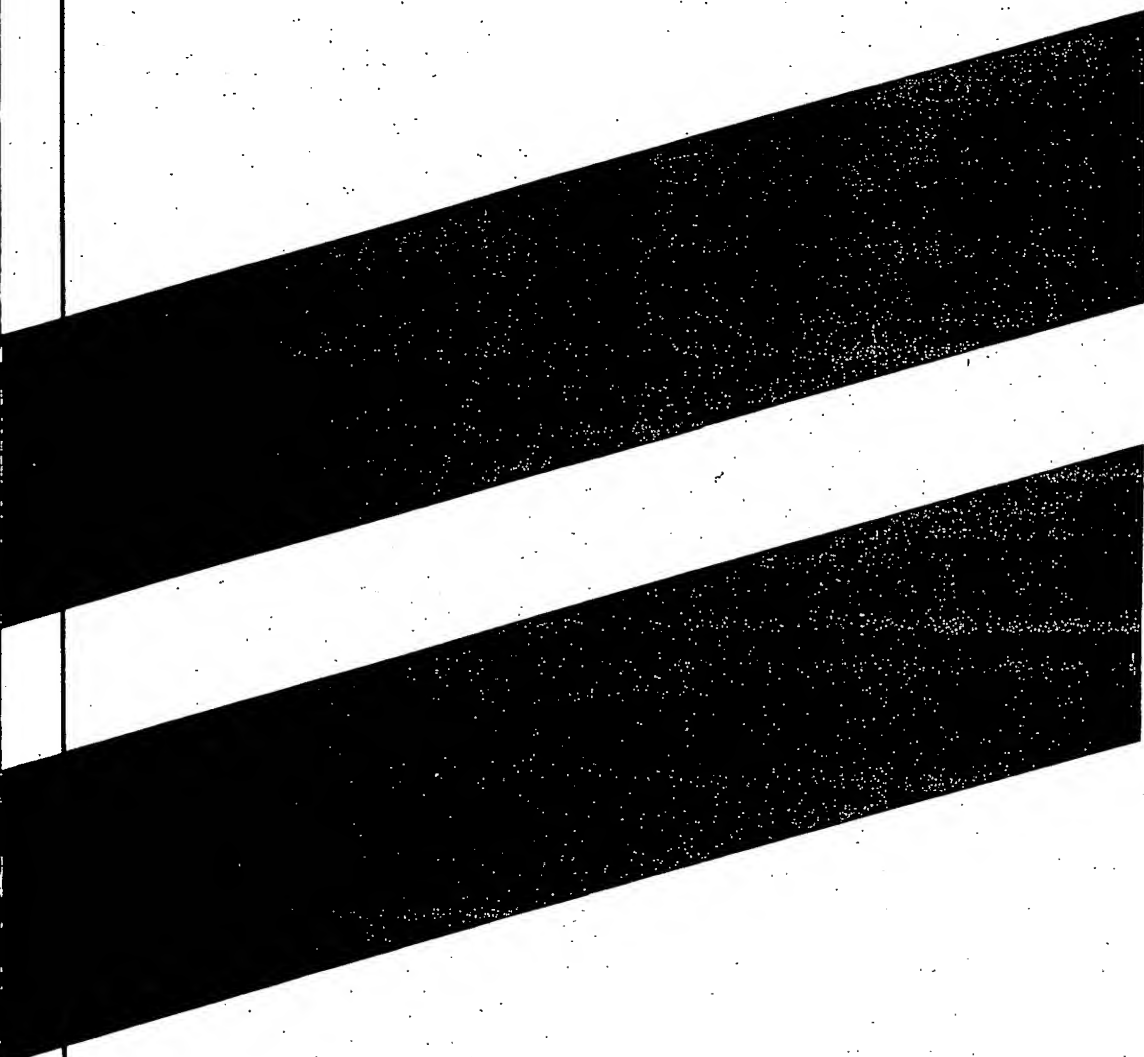
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PROVISIONAL
APPLICATION
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3.13-98



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SPEC. HAND	71622	9/9/98
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1c560 U.S. PTO

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PATENT APPLICATION



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Application _____ papers.

2. *CFR*

3. *Research*

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PATENT APPLICATION SERIAL NO. _____

U.S. DEPARTMENT OF COMMERCE
PATENT AND TRADEMARK OFFICE
FEE RECORD SHEET

13/19/1998 JRG/TTS 00000001 60077878
12-FC1214 75.00 CP

A TREATMENT SYSTEM FOR REMOVING IONIC CONTAMINANTS
FROM COPPER ELECTROLYTE SOLUTION

5

FIELD OF THE INVENTION

The present invention relates generally to a leaching process and specifically to a leaching process in which the pregnant leach solution is subjected to nanofiltration.

10

SUMMARY OF THE INVENTION

These and other needs are addressed by the metal recovery process of the present invention. The metal recovery process includes the steps of:

(a) contacting a leach solution with a metal-containing material to form a pregnant leach solution containing at least a portion of the metal and a contaminate metal;

(b) forming the pregnant leach solution into an electrolytic solution;

(c) contacting the electrolytic solution with an electric current to remove at least a portion of the metal and form a lean electrolytic solution including a portion of the metal and the contaminate metal;

(d) filtering at least a portion of the lean electrolytic solution with a filter to form a permeate containing at least most of the metal (and preferably a minority of the contaminant) in the lean electrolytic solution and a concentrate containing at least most of the contaminate

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metal (and preferably a minority of the metal) in the lean electrolytic solution; and

(c) recovering the metal from the permeate.

A variety of metals can be recovered by this process.

5 Most preferred metals are selected from the group consisting of copper, cobalt, nickel, and mixtures thereof. These metals are commonly present in the pregnant leach solution as a metal sulfate.

10 The contaminate metals can detrimentally affect the efficiency of the electrolytic recovery of the metal, particularly in high concentrations of the contaminate metals. Such metals include iron, manganese, lead, other tri-valent ions, and other ion complexes that are larger than the metal sulfate complex.

15 Filtration can be performed using a variety of ultrafiltration or nanofiltration membranes. Preferably, the filter has a pore size ranging from about 5 to about 500 angstroms and more preferably from about 10 to about 100 angstroms. Filtration is discussed in detail in U.S. Patents
20 5,116,511; 5,310,486; and 5,476,591, which are incorporated herein by reference fully in their entireties.

Filtration is conducted such that as much as possible of the electrolytic solution is contained in the permeate. The concentrate preferably comprises no more than about 95% of the
25 electrolytic solution and more preferably no more than about

50% of the electrolytic solution. In contrast, the permeate preferably comprises at least about 5% of the electrolytic solution and more preferably at least about 50% of the electrolytic solution.

5 Filtration removes a substantial amount of the contaminate metals from the permeate and places them in the concentrate. Preferably, the contaminant metals are reduced by at least about 5% and more preferably by at least about 25% in the permeate. In contrast, the concentrate preferably
10 comprises at least about 105% and more preferably at least about 125% of the contaminate metals levels in the electrolytic solution.

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 The pregnant leach solution can be formed into an electrolytic solution by (a) contacting the pregnant leach
15 solution with an organic solvent to form a copper rich organic solvent containing most of the copper and a copper lean leach solution that is substantially free of copper (which is recycled to the heap), (b) contacting the copper rich organic solvent with the copper lean electrolytic solution to form a
20 copper lean organic solvent that is substantially free of copper (which is recycled to the contacting step (a)) and the copper rich electrolytic solution that contains most of the copper in the copper rich organic solvent. The concentrate can be recycled to contacting step (b) for stripping the metal
25 from the copper rich organic solvent.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a flow schematic of a preferred embodiment according to the present invention.

5

DETAILED DESCRIPTION

Currently, build-up of ionic contaminants in the electrolyte of a copper electrowinning process is prevented by discharging a continuous bleed stream of lean electrolyte. This bleed stream volume has to be replaced, requiring
10 continuous quantities of acid and clean water. More importantly, the bleed stream removes a substantial amount of copper (and expensive cobalt additive) from the electrolyte circuit.

Iron, manganese, other tri-valent ions, and/or ion
15 complexes larger than the copper sulfate complex (i.e. contaminants) can be removed with a treatment system utilizing ultrafiltration or nanofiltration membranes. The copper containing solution, i.e. lean "electrolyte" from a copper electrowinning process, is passed through an ultrafiltration
20 or nanofiltration membrane system to produce a concentrate containing the majority of the ionic contaminants and a permeate containing a minority of the ionic contaminants. The concentrate is discharged as a small bleed stream to the PLS raffinate or to waste. The permeate is returned to the head
25 of the copper electrowinning process for copper recovery. The

membrane filtration of the copper electrolyte results in reduced bleed stream volumes, immediate recovery of additional copper in the EW, reuse of an expensive cobalt metal additive, and overall lower SX/EW operating costs.

5 The present invention generally relates to the removal of copper ions from copper ore, and more particularly enhanced removal of copper ions using an ultrafiltration or nanofiltration process which removes ionic contaminants from copper electrolyte providing an enhanced electrolyte for
10 electrowinning of copper ions.

 The techniques used to remove copper from raw ore determine the overall efficiency of the copper mining operation. Hydrometallurgical copper mining operations using a leaching system and a copper extraction plant, such as a
15 solvent extraction/electrowinning (SX/EW) plant, are now accepted processes in the copper mining industry. Currently, electrowon copper accounts for about 30% of total U.S. copper production. The industry trend continues toward
20 hydrometallurgical technology as higher-grade ores are depleted and smelting costs increase. Other advantages of hydrometallurgical technology, such as the ability to process low-grade ores, low labor requirements, ease of operation in remote areas, and low operating costs, make it attractive to mining companies.

5 "Copper hydrometallurgy," in which copper ions are leached or otherwise extracted from raw ore using liquid chemical agents, has been of interest since as early as the 17th century when copper recovery methods involving iron precipitating agents from sulfuric acid based copper solutions were tested. The hydrometallurgical circuit consists of copper leaching and copper recovery.

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10 Referring to Fig. 1, a copper leaching agent or "lixiviant" 10 is selected for use in leaching the desired metal (e.g., copper) ions from the desired metal-containing ore. Representative lixiviants include but are not limited to sulfuric acid (H_2SO_4), a combination of H_2SO_4 and ferric sulfate, $Fe_2(SO_4)_3$ (primarily for sulfide-containing ore materials), acidic chloride solutions (e.g. ferric chloride, 15 $FeCl_3$) or cupric chloride, $CuCl_2$), nitrate solutions, ammonia, and ammonium salt compositions. Sulfuric acid is by far the most common lixiviant. The lixiviant is applied to the ore 14 (which is stacked or piled in a large heap or dump) via a sprinkler type system and allowed to percolate downwardly into 20 the ore. As a result, desired metal ions are leached from the ore and collected within the lixiviant to generate a lixiviant product 18 that consists of a metal ion concentration (also known as a "pregnant leach solution"). The lixiviant exits the bottom of the ore and is collected to form the pregnant 25 leach solution 18. Further information regarding the

lixiviant leaching process is disclosed in U.S. Patent no. 5,476,591 to Green et al., which is incorporate herein by reference.

Next, a copper recovery process is used to selectively
5 extract copper from the collected lixiviant. Representative copper recovery processes include but are not limited to solvent extraction/electrowin (SX/EW), direct electrowinning, ion exchange - electrowin (IX/EW), and iron precipitation. Solvent extraction/electrowin is presently the most common
10 copper recovery process. SX/EW technology was implemented in the 1960s with the development of special organic extractants for copper. The SX/EW process consists of three closed solution loops. In the first loop 22, the pregnant leach solution 18 containing desired metal ions and a multitude of
15 other metal ions is fed into a mixer/settler tank 26 where it is contacted with a copper-extracting organic liquid, commonly referred to as a "lix". The "lix" 30 preferentially extracts from 70% to 90% of the desired metal ions from the pregnant leach solution 18 into a loaded organic 38. The second closed
20 loop 34 involves contacting the loaded organic 38 with a lean electrolyte stream 42 from the electrowinning process. The desired metal ions are transferred from the organic solution or "lix" 38 to the lean electrolyte 42 to form a loaded electrolyte 46. In the third and final closed loop 50, the
25 rich electrolyte flows between a cathode plate and an

insoluble anode, where 70% - 90% of the desired metal is removed through "plating." The electrochemical cell "plates" a stainless steel electrode with the desired metal using an applied current. The metal-plated cathode plates are then
5 periodically removed from the process to obtain the solid, high-purity metal product.

The metal leaching - metal recovery process must be improved to overcome inherent problems such as build-up of undesired "spectator" ions, particularly in the electrolyte
10 closed loop circuit. In a problem common to all mining operations, the organic extractants or "lix" chemicals are not completely selective in the metals they extract from the pregnant leach solution(PLS). For example, some iron loads onto the lix along with the desired metal. The iron is then
15 released into the electrolyte (or stripping) circuit along with the desired metal. In addition to ion contaminant transfer from PLS to electrolyte due to poor organic selectivity, ion contaminants can be transferred from the PLS to the electrolyte due to entrainment of PLS in the organic,
20 with subsequent release of the PLS into the electrolyte. Entrainment and organic selectivity are the main reasons for build-up of iron, manganese, ad other ionic contaminants in the electrolyte circuit.

The present invention specifically provides an improved
25 method for metal recovery in which a majority of the undesired

metals are removed from the electrolyte circuit, leaving a cleaner electrolyte stream for enhanced electrowinning. Accordingly, the invention represents an advance in the art of metal mining technology, as described in detail herein.

5 The process improvements claimed by the present invention will result from utilizing an ultrafiltration or nanofiltration membrane system 54 to process a bleed stream 58 of the lean electrolyte 42 exiting from the electrowinning plant. As described above and shown in Fig. 1, the lean
10 electrolyte 42, a strong sulfuric acid solution containing desired metal ions, is returned to the stripping stage to contact the loaded organic 30. Before entering the stripping stage, a small bleed stream 58 of lean electrolyte is removed, and replaced with a stream consisting of clean sulfuric acid
15 and water. This bleed stream, containing certain concentration of undesirable metal ions, is set at a rate equal to the total amount of undesirable metal ions entering the rich electrolyte from entrainment or organic transfer.

20 The "lean electrolyte bleed stream" is passed through the ultrafiltration or nanofiltration membrane system 54. The ultrafiltration or nanofiltration membrane 54 preferentially rejects higher percentages of iron, manganese, other tri-valent ions, and/or ion complexes larger than the copper sulfate complex (ion contaminants). The membrane system
25 separates the lean electrolyte into two streams: permeate and

concentrate. The concentrate stream consists of the majority of the ionic contaminants in the electrolyte because of the preferential rejection of ions larger than the copper sulfate complex, as mentioned above. The permeate stream consists of
5 a minority amount of the ionic contaminants.

The permeate stream, which comprises approximately 50% of the membrane feed, reports back to the electrolyte circuit. The concentrate stream, which comprises approximately 50% of the membrane feed, is discharged to the raffinate stream or
10 pond for reuse in ore leaching.

Example 1

In a recent field test of this apparatus, an electrolyte containing ionic contaminants was split into two streams as follows:

15		<u>Feed</u>	<u>Permeate</u>	<u>Rejection</u>
	Cu	2.44 g/L	2.0 g/L	18%
	Fe	3040 ppm	1920 ppm	37%
	Mn	36.4 ppm	28.9 ppm	21%

20 The primary ionic contaminant, iron, was rejected at a ~20% higher rate than copper. Discharging the membrane concentrate allows a significant reduction in bleed stream volume to maintain the same total amount of iron discharged from the electrolyte system.

Example 2

In another recent field test of this apparatus, a different electrolyte containing ionic contaminants was split into two streams as follows:

	<u>Feed</u>	<u>Permeate</u>	<u>Rejection</u>
Cu	34.2 g/L	26.5 g/L	22.5%
Fe	1.94 g/L	1.05 g/L	46%
Mn	79 ppm	61 ppm	23%
Co	172 ppm	142 ppm	17%

10

The primary ionic contaminant, iron, was rejected at a ~24% higher rate than copper. Again, discharging the membrane concentrate allows a significant reduction in bleed stream volume to maintain the same total amount of iron discharged from the electrolyte system. In addition, cobalt, a valuable metal which is added to the electrolyte to assist in electrowinning, is rejected at a lower rate than copper. Therefore, a portion of the valuable cobalt metal reports with the permeate back to the electrolyte circuit. In the current system, all cobalt in the bleed stream reports to the raffinate, where it is lost forever in the ore heaps.

The membrane system in the invention would process a bleed stream of lean electrolyte from the electrowinning circuit tank house. The system would process 10 - 1,000 gallons per minute of electrolyte, with ~5%-75% of the feed flow becoming permeate product. Typical ultrafiltration

membranes used would be G series elements (G-5, G-10, G20) from Osmonics/Desalination Systems of Vista, CA, and typical nanofiltration membranes used would be DK series elements from Osmonics/Desalination Systems of Vista, CA. A typical system
5 would process 100 gpm of electrolyte through 30 each 8 inch spiral wound G-5 membrane elements. The system would split the feed flow into 50 gpm of permeate and 50 gpm of concentrate, with about a 20-25% difference in rejection between copper and iron.

10 Replacing an existing bleed stream with a membrane treated discharge stream could result in a capital cost payback time of less than 6 months. The operating cost credits used to calculate this payback include the operating cost of reprocessing the copper not sent to the raffinate
15 stream, and the operating cost of not having to supply the make-up cobalt, acid, and water returned to the electrolyte circuit in the membrane permeate stream.

What is claimed is:

1. A method for recovering metal from a metal-containing material, comprising:

5 (a) contacting a leach solution with the metal-containing material to form a pregnant leach solution containing at least a portion of the metal and a contaminate metal;

(b) forming the pregnant leach solution into an electrolytic solution;

10 (c) contacting the electrolytic solution with an electric current to remove at least a portion of the metal and form a lean electrolytic solution including a portion of the metal and the contaminate metal;

15 (d) filtering at least a portion of the lean electrolytic solution with a filter to form a permeate containing at least most of the metal in the lean electrolytic solution and a concentrate containing at least most of the contaminate metal in the lean electrolytic solution; and

(c) recovering the metal from the permeate.

2. The method of Claim 1, wherein the metal is selected from the group consisting of copper, cobalt, nickel, and mixtures thereof.

3. The method of Claim 1, wherein the contaminate metal is selected from the group consisting of iron, manganese, lead, other trivalent metals, and mixtures thereof.

4. The method of Claim 1, wherein the nanofilter has a pore size ranging from about 5 to about 500 Å.

5. The method of Claim 1, wherein the concentrate comprises no more than about 95% of the lean electrolytic solution and the ratio of contaminant metals to the metal in the concentrate has been increased by at least about 5% relative to the ratio of the lean electrolyte solution.

6. The method of Claim 1, wherein the permeate comprises at least about 5% of the lean electrolytic solution and the ratio of contaminant metal to the metal in the permeate has been decreased by at least about 5% relative to the ratio of the lean electrolyte solution.

7. The method of Claim 1, wherein the permeate contains less than about 50% of the contaminate metals in the lean electrolyte solution.

8. The method of Claim 1, wherein the concentrate comprises at least about 105% of the contaminate metals in the lean electrolytic solution.

9. The method of Claim 1, wherein the permeate comprises at least about 50% of the metal in the lean electrolytic solution.

10. The method of Claim 1, wherein the forming step comprises the substeps of:

(i) contacting the pregnant leach solution with an organic solvent to form a metal rich organic solvent containing at least most of the metal and a portion of the contaminate metal and a metal lean leach solution; and

(ii) contacting the metal rich organic solvent with the metal lean electrolytic solution to form a metal lean organic solvent and a metal rich electrolytic solution that contains at least most of the metal in the metal rich organic solvent.

11. The method of Claim 11, further comprising:

(d) recycling the concentrate to step (ii).

12. The method of Claim 1, wherein the metal is in a metal complex and the contaminate metal is in a contaminate

metal complex and the metal complex has a smaller atomic size than the contaminate metal complex.

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VERIFIED STATEMENT (DECLARATION) CLAIMING SMALL ENTITY STATUS
(37 CFR 1.1(f) and 1.27(c)) - SMALL BUSINESS CONCERN

I hereby declare that I am an official empowered to act on behalf of the small business concern, HW PROCESS TECHNOLOGIES, INC. of 1208 Quail Street, Lakewood, Colorado 80215.

I hereby declare that the above-identified small business concern qualifies as a small business concern as defined in 13 CFR 121.3-18, and reproduced in 37 CFR 1.9(d), for purposes of paying reduced fees under section 41(a) and (b) of Title 35, United States Code, in that the number of employees of the concern, including those of its affiliates, does not exceed 500 persons. For purposes of this statement, (1) the number of employees of the business concern is the average over the previous fiscal year of the concern of the persons employed on a full-time, part-time or temporary basis during each of the pay periods of the fiscal year, and (2) concerns are affiliates of each other when either, directly or indirectly, one concern controls or has the power to control the other, or a third party or parties controls or has the power to control both.

I hereby declare that rights under contract or law have been conveyed to and remain with the small business concern identified above with regard to the invention, entitled "A TREATMENT SYSTEM FOR REMOVING IONIC CONTAMINANTS FROM COPPER ELECTROLYTE SOLUTION" and identified as Attorney File No. 3376-28PROV, described in the specification filed herewith.

If the rights held by the above-identified small business concern are not exclusive, each individual, concern or organization having rights to the invention is listed below* and no rights to the invention are held by any person, other than the inventor, who could not qualify as a small business concern under 37 CFR 1.9(c) or by any concern which would not qualify as a small business concern under 37 CFR 1.9(d) or a nonprofit organization under 37 CFR 1.9(e).

*NOTE: Separate verified statements are required from each named person, concern or organization having rights to the invention averring to their status as small entities. (37 CFR 1.27)

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ADDRESS _____
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I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR 1.28(b))

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

Date: 1-21-98

By: Harold E. Whatley
Harold E. Whatley
Vice President Finance and Administration
1208 Quail Street
Lakewood, Colorado 80215

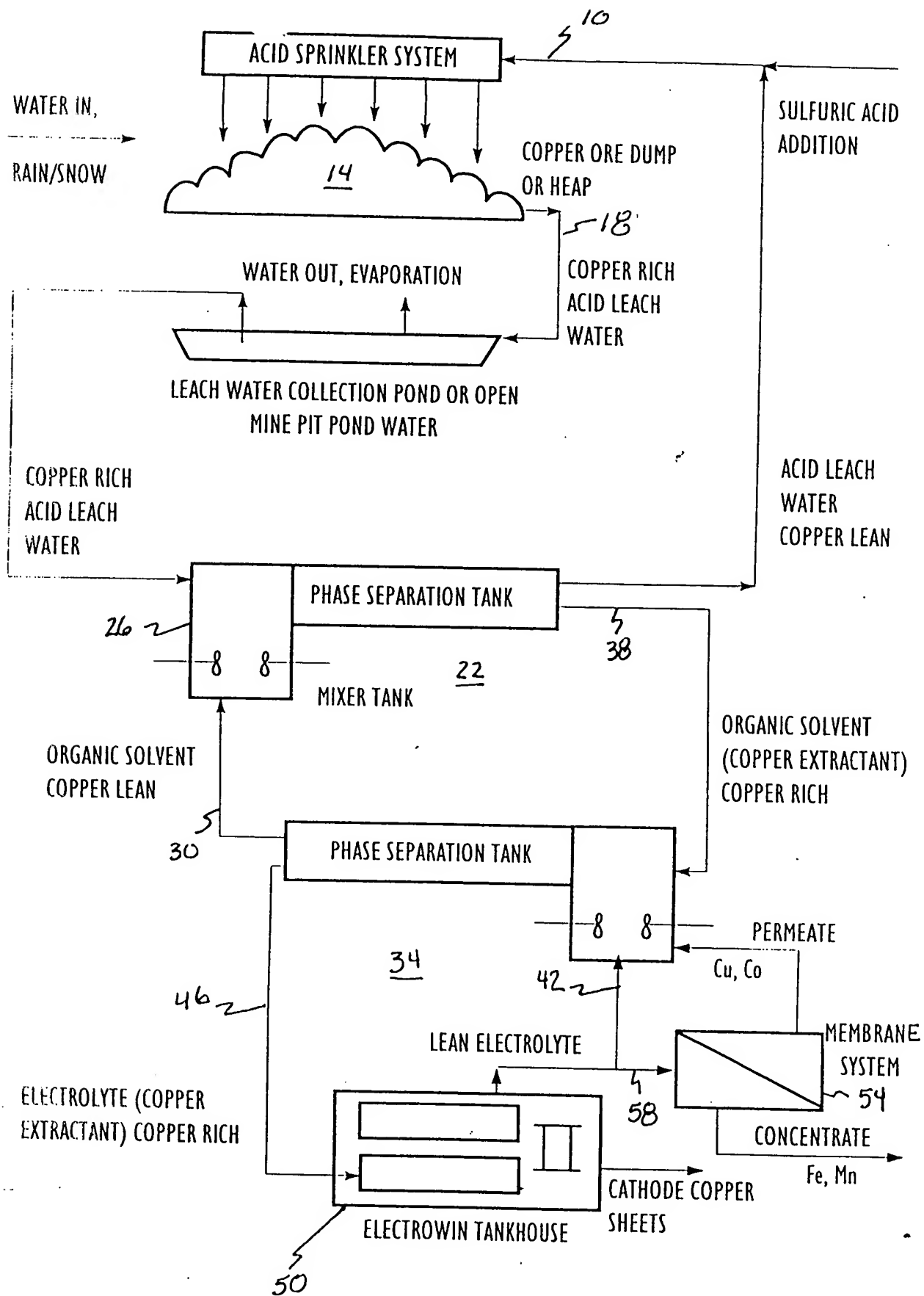


FIG. 1



UNITED STATES DEPARTMENT OF COMMERCE
Patent and Trademark Office

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LOCATION 9200 SERIAL NUMBER 60077878 PATENT NUMBER

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PROVISIONAL APPLICATION COVER SHEET

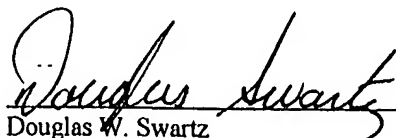
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This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(b)(2).

Docket Number		3376-28PROV		Type a plus sign (+) inside this box -	+
INVENTOR(s)/APPLICANT(s)					
LAST NAME	FIRST NAME	MIDDLE INITIAL	RESIDENCE (CITY AND EITHER STATE OR FOREIGN COUNTRY)		
Green	Dennis	H.	9455 West 68th Avenue Arvada, Colorado 80004		
Mueller	Jeff		3462 Cripple Creek Square Boulder, Colorado 80303		
TITLE OF THE INVENTION (280 characters max)					
"A TREATMENT SYSTEM FOR REMOVING IONIC CONTAMINANTS FROM COPPER ELECTROLYTE SOLUTION"					
CORRESPONDENCE ADDRESS					
Douglas W. Swartz, Esq. Sheridan Ross P.C. 1700 Lincoln Street, Suite 3500 Denver					
STATE	Colorado	ZIP CODE	80203	COUNTRY	United States of America
ENCLOSED APPLICATION PARTS (check all that apply)					
X	Specification	Number of Pages	16	X	Small Entity Statement
X	Drawing(s)	Number of Sheets	1		Other (specify)
METHOD OF PAYMENT (check all that apply)					
X	A check or money order is enclosed to cover the filing fees			PROVISIONAL FILING FEE AMOUNT (\$)	\$75.00
X	The Commissioner is hereby authorized to charge filing fees and credit Deposit Account Number:			19-1970	

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

- ☒ No.
- ☐ Yes, the name of the U.S. Government agency and the Government contract number are: _____


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Date: March 13, 1998

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REQUEST FOR ACCESS OF ABANDONED APPLICATION UNDER 37 CFR 1.14(a)

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Application Number

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60/077,878

3-13-98

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Assistant Commissioner for Patents
Washington, DC 20231

I hereby request access under 37 CFR 1.14(a)(3)(iv) to the application file record of the above-identified ABANDONED application, which is: (CHECK ONE)

- ☒ (A) referred to in United States Patent Number 6176186 column _____
- ☐ (B) referred to in an application that is open to public inspection as set forth in 37 CFR 1.11, i.e., Application No. _____ filed _____ on page _____ of paper number _____
- ☐ (C) an application that claims the benefit of the filing date of an application that is open to public inspection, i.e., Application No. _____ filed _____ or
- ☐ (D) an application in which the applicant has filed an authorization to lay open the complete application to the public.

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Unit: F-1-4



US006156186A

United States Patent [19]

Mueller et al.

[11] Patent Number: 6,156,186

[45] Date of Patent: *Dec. 5, 2000

[54] METHOD FOR REMOVING
CONTAMINANTS FROM PROCESS
STREAMS IN METAL RECOVERY
PROCESSES[75] Inventors: Jeff Mueller, Boulder; Dennis H.
Green, Arvada, both of Colo.[73] Assignee: HW Process Technologies, Inc.,
Lakewood, Colo.[*] Notice: This patent is subject to a terminal dis-
claimer.

[21] Appl. No.: 09/183,683

[22] Filed: Oct. 30, 1998

Related U.S. Application Data

[60] Provisional application No. 60/100,510, Sep. 16, 1998,
provisional application No. 60/100,494, Sep. 16, 1998,
provisional application No. 60/077,878, Mar. 13, 1998,
provisional application No. 60/077,428, Mar. 9, 1998, pro-
visional application No. 60/064,284, Oct. 30, 1997, provi-
sional application No. 60/064,279, Oct. 30, 1997, provi-
sional application No. 60/099,717, Sep. 10, 1998, and
provisional application No. 60/100,497, Sep. 16, 1998.[51] Int. Cl.⁷ C25C 1/00; C25C 1/16;
C25C 1/20; C15G 1/12; B01D 11/00[52] U.S. Cl. 205/560; 205/568; 205/569;
205/570; 205/571; 205/581; 205/582; 205/605;
205/606; 210/650[58] Field of Search 205/560, 568,
205/571, 580, 589, 594, 569, 570, 581,
582, 590, 591, 605, 606; 210/650

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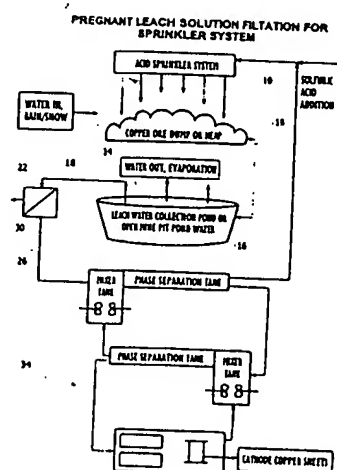
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[57] ABSTRACT

The present invention is directed to a process for removing
various contaminants (e.g., organic collectors, contaminant
metals or spectator ions, and/or suspended and colloidal
solids) from process streams in leaching processes. The
contaminant removal is performed by one or more mem-
brane filtration systems (e.g., nanofilters, ultrafilters, and/or
microfilters) treating process streams including, the pregnant
leaching solution, the barren raffinate, and the lean and rich
electrolytes.

33 Claims, 7 Drawing Sheets



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APPLICANT

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****CONTINUING DOMESTIC DATA*******
 VERIFIED

****371 (NAT'L STAGE) DATA*******
 VERIFIED

****FOREIGN APPLICATIONS*******
 VERIFIED

FOREIGN FILING LICENSE GRANTED 05/20/98 ***** SMALL ENTITY *****

Foreign Priority claimed <input type="checkbox"/> yes <input type="checkbox"/> no 35 USC 119 (a-d) conditions met <input type="checkbox"/> yes <input type="checkbox"/> no <input type="checkbox"/> Met after Allowance Verified and Acknowledged _____ <u>Examiner's Initials</u> <u>Initials</u>	STATE OR COUNTRY CO	SHEETS DRAWING 1	TOTAL CLAIMS	INDEPENDENT CLAIMS
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TITLE

TREATMENT SYSTEM FOR REMOVING IONIC CONTAMINANTS FROM COPPER
 ELECTROLYTE SOLUTION

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